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TECHNICAL REPORT BRL-TR-3329

BRL

SHOCK TUBE STUDY OF THE REACTION OF TRIETHANOL AMMONIUM NITRATE WITH N_2O

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RICHARD A. BEYER

APRIL 1992

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	April 1992	Final, Sep 89 - Dec 90

4. TITLE AND SUBTITLE

Shock Tube Study of the Reaction of Triethanol Ammonium Nitrate With N_2O

5. FUNDING NUMBERS

PR: 1L161102AH43

6. AUTHOR(S)

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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

U.S. Army Ballistic Research Laboratory
ATTN: SLCBR-DD-T
Aberdeen Proving Ground, MD 21005-5066

10. SPONSORING / MONITORING AGENCY REPORT NUMBER

BRL-TR-3329

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION / AVAILABILITY STATEMENT

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12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

The ignition of triethanol ammonium nitrate in O_2 and N_2O has been studied in a shock tube over the temperature range from 1,300 K to 1,950 K. The ignition threshold for TEAN in 5% N_2O is above 1,300 K. The reactions can be described by Arrhenius kinetics with activation energies of 17.0 kcal/mol and 20.7 kcal/mol, respectively.

14. SUBJECT TERMS

liquid propellants, LP1846, shock tube, triethanol ammonium nitrate (TEAN), kinetics

15. NUMBER OF PAGES

21

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAR

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TABLE OF CONTENTS

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LIST OF FIGURES

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1. INTRODUCTION

Liquid propellants based on aqueous solutions of hydroxyl ammonium nitrate (HAN) and various fuels have been the subject of extensive studies (Decker et al. 1987). Of particular interest are the propellants designated LP1845 and LP1846, where the fuel is triethanol ammonium nitrate (TEAN). In our earlier work (Beyer 1990) we have shown that nitrous oxide, known to be a major product of the thermal decomposition of HAN, reacts with (ignites) TEAN powder. In this study, we further characterize this reaction at lower pressures, and attempt to produce an overall kinetics rate constant for this complex reaction.

2. EXPERIMENTAL

The shock tube used in these studies is stainless steel with a 98-mm inside diameter. The driver section is 1.2 m long; the driven (test) section is 6.3 m long. For typical conditions of the present measurements, a 0.001-in (25.4-μm) thick Mylar polyester diaphragm was used. The initial pressures were 18 psi (120 kpa) in the driver section and from 5 to 20 torr (0.6 to 2.6 kpa) in the test section. The test gas was either 5% oxidizer in argon or pure argon. The driver gas was 30% air with the balance helium to maximize the test time. Under typical conditions, the passage of the initial shock raises the test gas to a temperature of about 750–1,000 K with a corresponding pressure increase. The reflected shock typically doubles the temperature of the gas and also raises the pressure. These present measurements were carried out over a range of reflected shock temperatures from 1,300 K to 1,900 K. Pressure in the reflected shock was near one half an atmosphere. The pressures were lower than those of the earlier studies in an attempt to increase the ignition delay time to where the temperature dependence could be more easily determined.

The layout of the test end of the shock tube is shown in Figure 1. A variety of instrumentation was used. Pressure transducers were placed at a minimum of two points to characterize the shock wave propagation. From the calculated shock velocity, a frozen-chemistry thermochemical equilibrium code (Gordon and McBride 1971) was used to calculate the pressure and temperature behind the shock waves. Ignition time was detected through a window in the end wall by a photomultiplier tube with a 389-nm filter, where strong CN radical emission from the onset of combustion is favored over other sources of light in the tube. A PbSe infrared detector with appropriate wavelength filters was used to study the emission from the nitrous oxide. In some experiments, carbon dioxide emission from mixtures of 5%

carbon dioxide in argon were also studied to compare signal loss for a molecule not expected to dissociate as rapidly as the nitrous oxide. The field of view for the infrared studies was a narrow region near pressure gauge No. 1 in Figure 1 (viewed from the side). A limited number of observations were also made with a Fairchild model 1200C line camera to verify that the ignition was taking place as predicted. In our present setup, we were able to record a line image with 200-point spatial resolution each 50 μs. All signals were recorded with a 4-channel transient digitizer and analyzed with an i487-based ("PC-clone") desktop computer.

The sample of TEAN to be ignited (typically < 20 mg) was ground to a fine powder and placed on a 1-in-diameter disk with feathered edges mounted in the center of the tube about 210 mm from the end wall. TEAN is fairly hygroscopic. Samples were kept in a desiccator prior to use, and further outgassed in the evacuated shock tube for about 1 hour on humid days and when it was thought possible that water may have been added during transfer to the shock tube. No attempt was made to analyze the percent hydration of the samples. During a shock experiment, the incident shock wave sweeps the powder off the disk and disperses it in the region between the disk and the end wall. It is assumed that the smallest particles, which are the earliest to ignite, move with the gas velocity behind the shock wave. The incident shock is not sufficiently hot in these experiments to ignite the sample and its chemical effect is ignored. The reflected shock returns from the end wall, heats the particles, which then ignite after a measured delay time in the stagnant gas. The smallest particles are calculated to be on the order of 100 mm from the end wall when they encounter the reflected shock. The pressure and shock transit time between pressure gauges are measured directly. The pressure rise associated with the shocks is calculated from the code (Gordon and McBride 1971); as long as the calculated and measured pressures are in reasonable agreement, the experimental behavior is assumed to be ideal and the calculated numbers are used to determine the time of interaction of the reflected shock and the smallest TEAN particles.

3. OBSERVATIONS

A series of line images from the line camera is shown in Figure 2. These images were recorded under somewhat stronger shock conditions than typical data in order to raise the temperature and provide sufficiently strong signals for the camera to record. As can be seen in the figure, there is a small amount of early light at approximately 25 mm (12 arb units) from the end wall, followed by ever greater amounts of light as the larger particles to the right ignite and burn. In subsequent images, the amount of light grows even greater to the right for about a millisecond. Because of experimental difficulty and variability,

the camera pixel units were not calibrated; rather, the predicted point of ignition was marked with a light source and compared to the observation. The total field of view of the camera was on the order of 4 inches (100 mm). The apparent signal peak at the end wall has not been addressed in detail; it may be due to impurities.

An important issue raised in our earlier study was on the thermal stability of the nitrous oxide. Before pursuing these ignition measurements further, a study of this stability was made using the infrared detector and a 4.49-μm bandpass filter. Without the filter, the emission followed the pressure trace very well (but with a much slower rise time). A typical trace of good behavior observed with the filter is shown in Figure 3a. The incident and reflected shocks pass the point of observation at 0 and 200 μs, respectively. The relatively slow rise (compared to the pressure, which is instantaneous on this time scale) is assumed to be instrumental. As can be seen, the N_2O emission was probably not affected by thermal decomposition before the ignition time; this held true as long as we stayed below about 1,900 K. An example of a hotter shock and presumed loss of N_2O due to thermal decomposition is shown in Figure 3b. Under some conditions the 4.49-μm signal was not as steady as expected based on temperature and the pressure records. Under these conditions, measurements were made with a mixture of CO_2 in argon and a 4.29-μm filter. The same unsteady behavior and limited test time were observed, leading to the conclusion that the temperature variations were flow-related; shock tube conditions, especially pressure ratios, were adjusted accordingly to minimize these changes.

The ignition was determined from the light detected through the 389-nm filter, which is assumed to be dominated by CN emission. There are clearly other sources of light at this wavelength under the conditions of these experiments. However, as long as our tube was kept reasonably clean, in order to limit the amount of black body emission from background particles, the ignition discrimination was reasonably good. Figure 4 shows the light signals detected with samples of TEAN shocked under nearly identical conditions ~ 1,600 K and 0.5 atm. As can be seen in the figure, the light rises much earlier with the N_2O present. In Figure 4, the two signals are not on the same scale; the quantity of light with an oxidizer present (e.g., N_2O in this case) is always much greater. For comparison, measurements were also made with oxygen/argon mixtures. The ignition characteristics were somewhat different, as shown in Figure 5. Of particular note is the indication of a distinctive slope break, which was frequently, but not always, present in the oxygen shots. This phenomenon will be discussed in more detail later. The oxygen shots were also different in that there was almost always total consumption of the TEAN powder, compared to the nitrous oxide shots where a considerable residue of the larger particles always remained. Although

it was not carefully quantified, the ignition threshold for TEAN in 5% N_2O/Ar under the conditions of these experiments was about 1,325 K.

4. ANALYSIS

As mentioned earlier, the shock wave calculations are assumed to describe the experimental conditions. In order to measure an ignition delay time, the time that the particles enter the hot flow must be known. The assumptions are made that the smallest solid particles move with the gas velocity behind the incident shock, and that these same small particles heat up on a time scale short compared with the delay time (i.e., instantaneously) and that they are the first to ignite. The ignition delay time of these smallest particles is thus calculated from the results of the shock wave calculations which are based on the velocity, which is determined from the pressure gauge timing measurements, and the initial test section pressure. Thus, time zero for the heating of the particles is the calculated time for the smallest particles to enter the reflected shock region.

In order to deduce a "first light" time from the photomultiplier signals, the derivative of the signal is taken. For most of the N_2O signals, this technique yields the same result as careful inspection of the light signal. However, for the O_2 observations, there is frequently, but not always, a second sharp break in the derivative.

It is reasonable to first assume that the kinetics are described by an Arrhenius form,

$$\text{Rate} = [\text{oxidizer}]Ae^{-Ea/RT} \quad ,$$

where [oxidizer] is the concentration of N_2O or O_2. In our measurements, the fraction of the gas which is oxidizer is always 5%, so this concentration is proportional to the pressure/temperature ratio in the reflected shock region. Since the observed delay time is proportional to the inverse of the rate, we can rewrite this expression as:

$$\frac{\text{Delay} * \text{Pressure}}{\text{Temperature}} = A' e^{Ea/RT} \quad .$$

Graphically, if we plot the natural log of the product of the delay time with the (calculated) pressure divided by the (calculated) temperature vs 1,000/temperature, the slope is E_a/R where E_a is the activation energy of the reaction and R is the gas constant. Figure 6 shows such a plot for the ignition of TEAN by N_2O/Ar. Although the number of data is not large, the behavior is reasonable. Since there are two breaks in the oxygen data, both were plotted and analyzed (Figure 7). In order to determine the activation energy more accurately, the data were fit to the full Arrhenius form using the well known but undocumented FITTER program of A. J. Kotlar. The activation energy from these N_2O data is 20.7 ±1.1 kcal/mol where the uncertainty is one standard deviation. The activation energy for the oxygen data analyses are 16.4 ±1.2 kcal/mol and 17.6 ±6.4 kcal/mol for the first and second breaks, respectively. These are essentially the same within their statistical uncertainties (standard deviations) and have been combined as one data set to give a value of 17.0 ±3.3 kcal/mol for the reaction with oxygen. It is of some note that the magnitude of the delays is not greatly different for the two oxidizers studied here.

5. DISCUSSION

The interpretation of the numbers measured here is interesting. When the TEAN is heated in the shock tube, it possibly can burn as solid; melt and burn as a liquid; or melt, thermally decompose, and burn as a gas. The melting point of TEAN is near 80° C with decomposition taking place at much higher temperatures—nearer 280° C. There was not a readily available apparatus for the measurement of the heat of fusion prior to the preparation of this report. A search of some previous contract work done by the Navy (Skahan 1987) yielded values of 149 J/g (7.55 kcal/mol) for pure TEAN and 292 J/g (14.8 kcal/mol) for 80% TEAN, with the balance assumed by the present author to be water of hydration of this hygroscopic material. As mentioned in Section 2, reasonable efforts were made to keep the TEAN dry, but no analysis was made. The measured values of 20.7 kcal/mol and 17.0 kcal/mol are strongly suggestive that, at least with oxygen, and possibly with nitrous oxide, we are measuring the melting kinetics and that the ignition reactions take place very rapidly after melting. If one concludes that the melting is the rate limiting step of this reaction, then the variability of these measured numbers may well be due to variations in the water content of samples used.

A second possibly important observation is that the oxygen consumes the sample completely whereas the nitrous oxide leaves many of the larger sized particles unaffected on the end and side walls of the shock tube. If both were rate limited by the melting of the sample, one might reasonably expect similar consumption of the sample in both cases. Thus, the difference in activation energy may in fact be real

and suggests that the nitrous oxide does not react as rapidly with the liquid TEAN as does oxygen. This might be significant in that in the liquid propellant combustion, the TEAN never exists as an isolated solid, since it is above its melting point before significant reaction takes place.

6. CONCLUSION

Measurements have been made of the activation energy for the reaction of TEAN with oxygen and nitrous oxide in argon. The magnitude of the numbers measured and other observations suggest that oxygen reacts rapidly with the TEAN after melting, while the nitrous oxide reaction may have a rate limiting step other than simple melting.

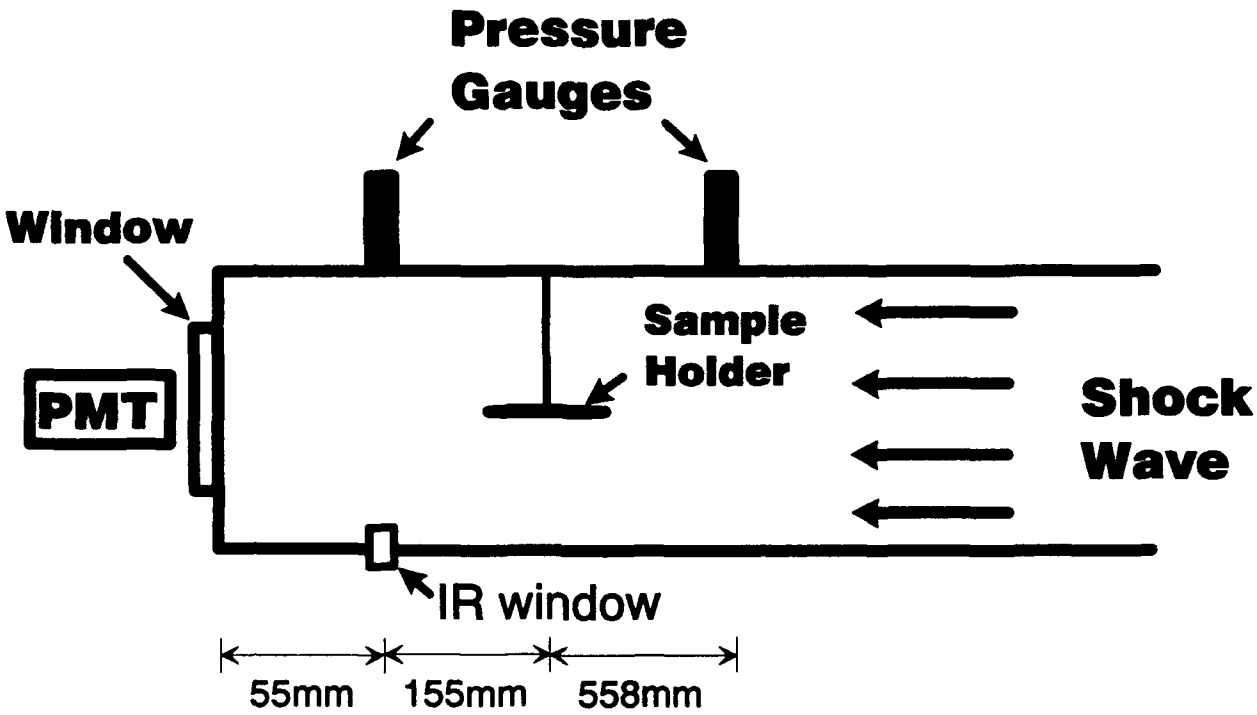


Figure 1. Schematic Diagram of the Test End of the Shock Tube.

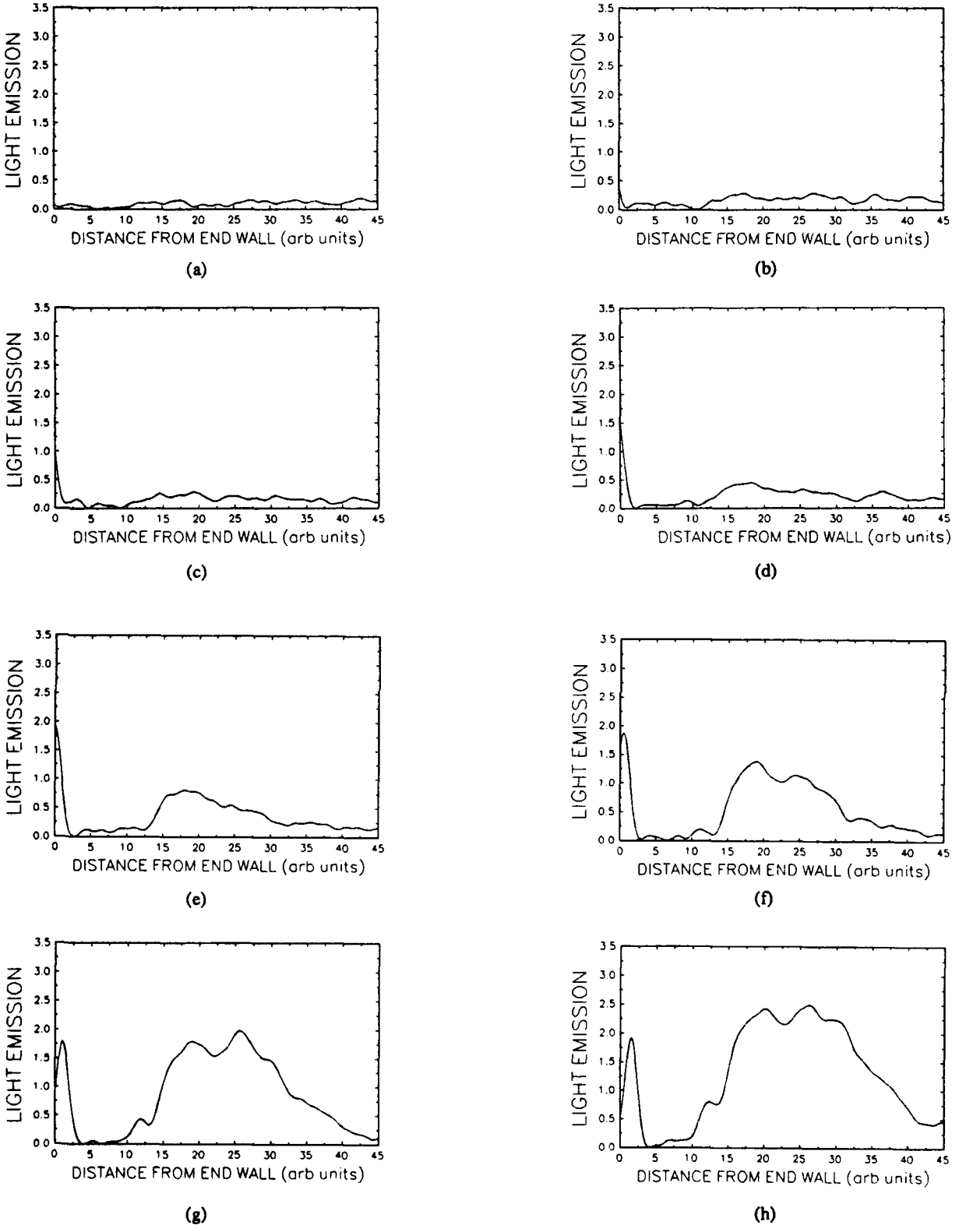
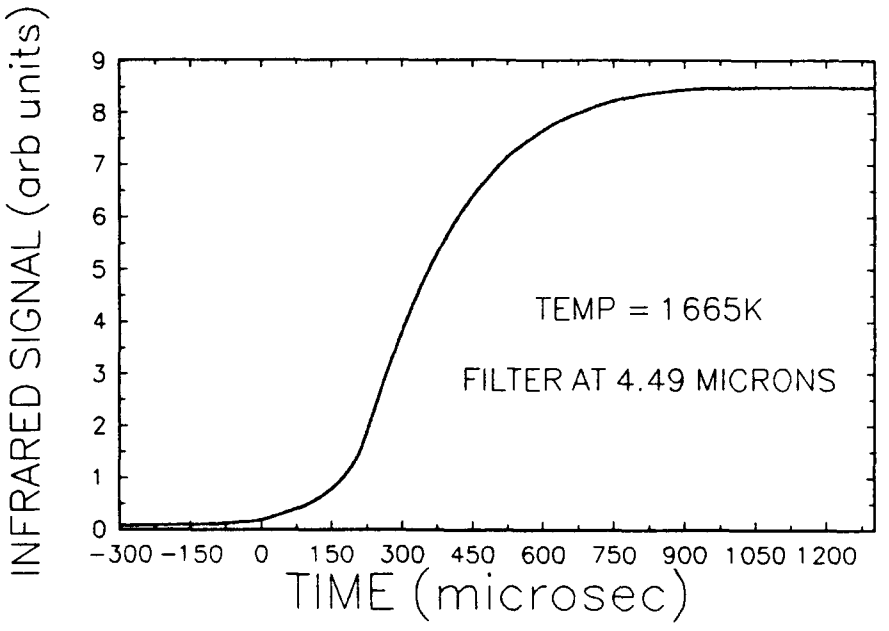
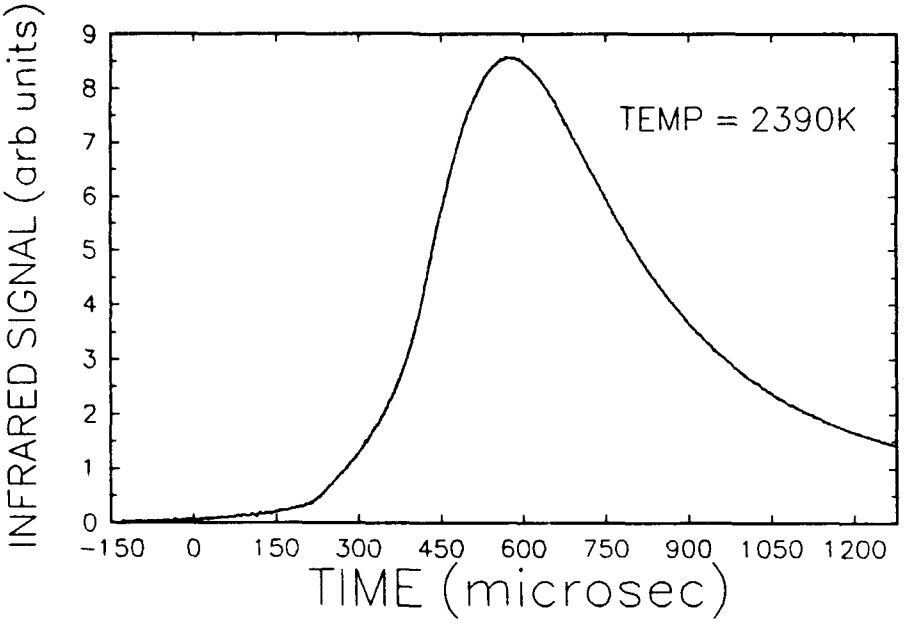


Figure 2. Eight Consecutive Line Images at 50-μs Intervals From the End of the Shock Tube During Ignition of TEAN in N_2O/Ar.



a. 1,665 K



b. 2,390 K

Figure 3. Signal From PbSe Infrared Detector at 4.49 μm (N,O Emission) During Passage of Shocks for Calculated Reflected Shock Temperatures.

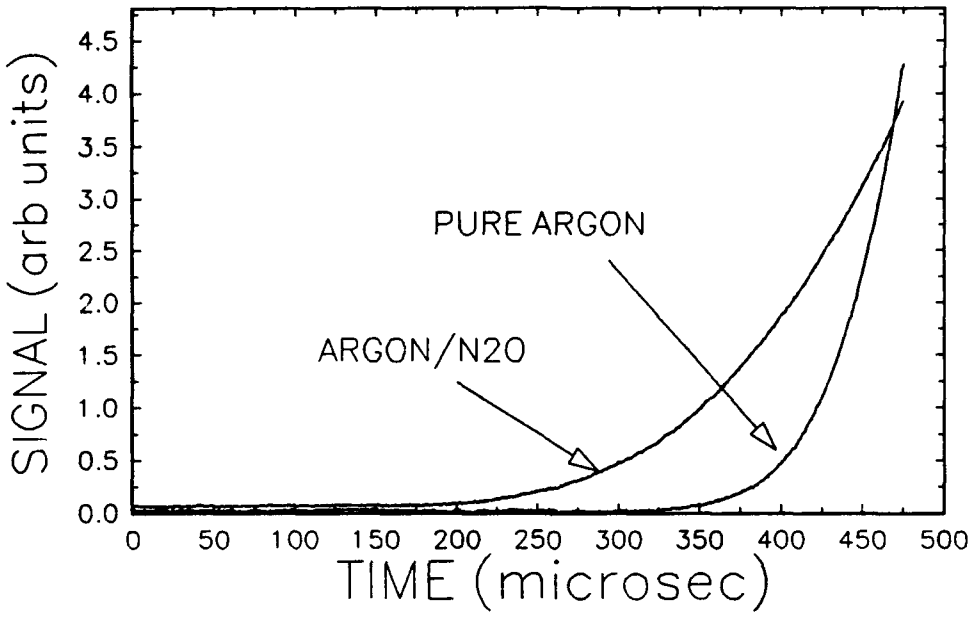


Figure 4. Light Signals Detected at 389 nm for TEAN Heated in N_2O/Ar and Pure Ar.

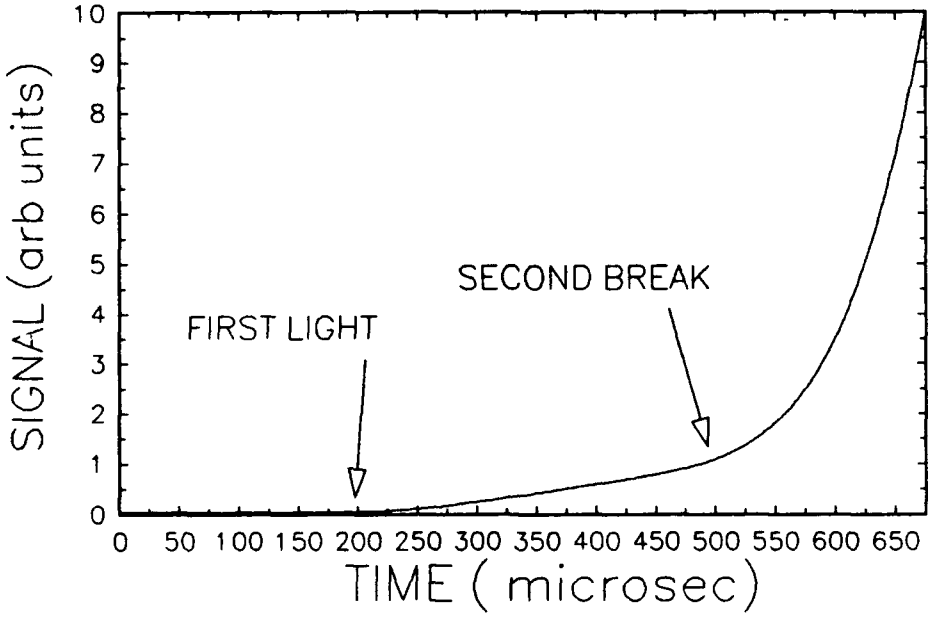


Figure 5. Light Detected at 389 nm for TEAN Heated in O_2/Ar.

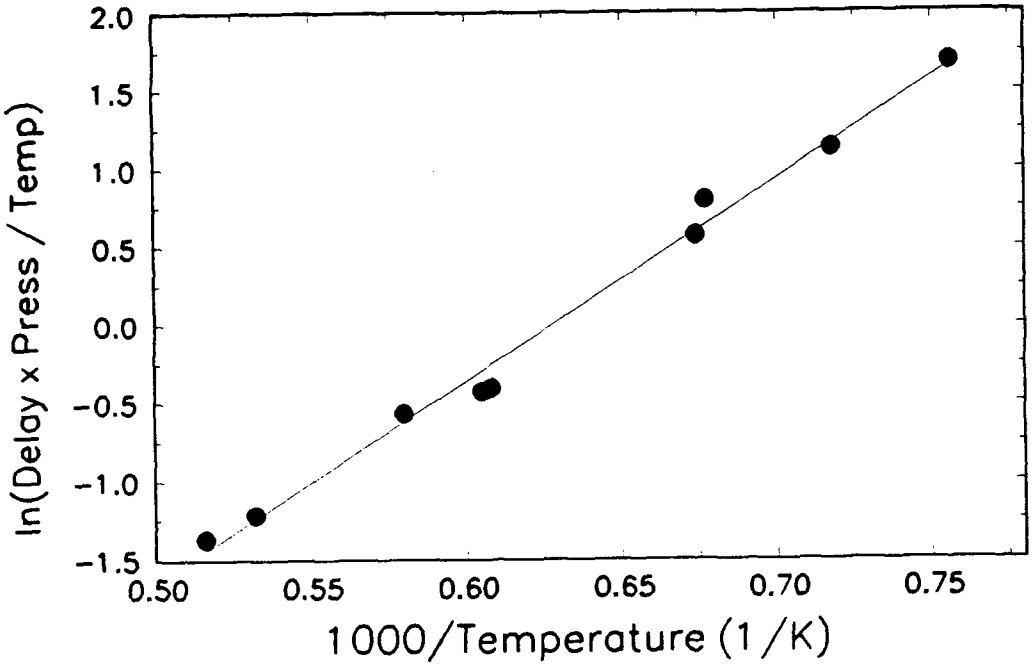


Figure 6. Arrhenius Plot of Ignition Delay vs. Inverse Temperature for TEAN Heated in N_2O/Ar.

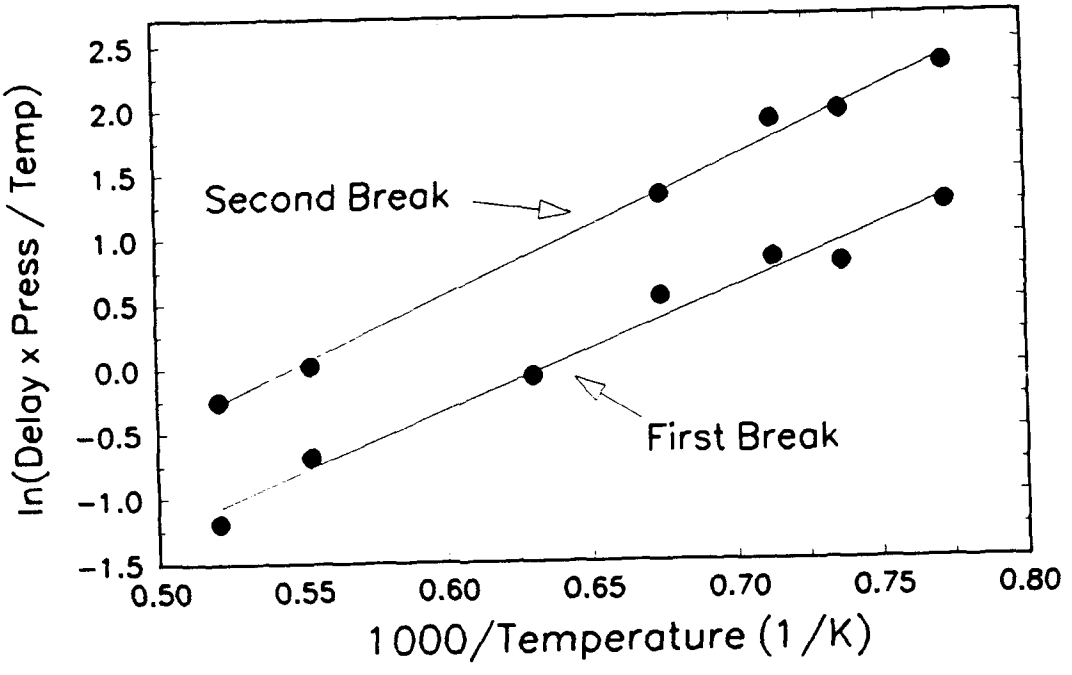


Figure 7. Arrhenius Plot of Ignition Delay vs. Inverse Temperature for TEAN in O_2/Ar.

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